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# Optimization of synthesis and characterization of nanosilica produced from rice husk (a common waste material)

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## Abstract

Rice husk (RH), an inexpensive waste material, was used to produce nanosilica. Acid treatment of RH followed by thermal combustion under controlled conditions gave 22.50% ash of which 90.469% was silica. Various chemical treatments in varied conditions for controlled combustion were investigated in order to produce highly purified nanosilica. The structural properties (such as X-ray diffraction, Brunauer-Emmett-Teller, Fourier transform infrared spectroscopy, and transmission electron microscopy) of the silica were studied. The method was optimized, and the chemical composition of the product was determined by X-ray fluorescence and carbon, hydrogen, and nitrogen analysis. Lime reactivity of the ashes was determined. At optimized conditions, a nanosized, highly purified silica (98.8 mass percentage) was produced with a high surface area, high reactivity, and 99.9% amorphous in form. Strength and number of acidic sites were measured by potentiometric titration. This nanosilica showed strong and a large number of acidic sites in comparison with commercial silica, making it as a good support for catalysts. This economic technology, as applied to waste material, also provides many benefits to the local agro-industry.

**Keywords:** Nanomaterial, Amorphous material, Nanosilica, Rice husk ash, Silica powder, Surface properties

## Background

Rice is one of the major food crops in the world. Its production generates an equally great amount of waste in the world, namely rice husk (RH), a by-product of the multistage processing of rice. RH accounts for approximately one-fifth of the annual gross rice production in the world, which currently stands at 545 million metric tons [1]. Rice production constitutes a dominant agro-industry worldwide, where rice is the main food crop that is grown in the waterlogged semitropical areas. With such a large availability of raw materials, it becomes economical to make them as sources of more commercial applications. Presently, having no commercial value in itself, RH usually ends up being burned in open spaces, thus causing environmental pollution and disposal problems. Due to the need to conserve energy and resources, efforts have been made to burn the husk under controlled conditions and to utilize the resultant ash as building, semiconductor, composite, and abrasive

materials [2-5]. Also, ash is an active catalyst and a good material for catalyst support because of its high surface area [6].

The chemical composition of RH has been found to vary from sample to sample. Any of the differences in type of paddy, crop year, climatic and geographical conditions, soil chemistry, sample preparation, or method of analysis could be a reason for this variation. RH is an excellent source of high-grade silica [7-9]. The presence of silica in RH has been known since 1938 [10], ever since several efforts on preparation of silica from RH by researchers have been known internationally [11-18]. This kind of silica has been shown to be a good material for the synthesis of very pure silicon, silicon nitride, silicon carbide, magnesium silicide, and other applications [6,9,19]. Recently, nanotechnology has aroused considerable scientific interest because of new potential uses of particles on a nanometric scale. Thus, industries may be able to reengineer many existing products to function at unique levels. The production of reactive nanoscale silica from RH is a simple process compared to other conventional production techniques such as vapor phase

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reaction, sol–gel process, etc. [6,20–22]. The emphasis of this paper is to optimize the conditions for the preparation of highly purified rice husk ash (RHA), of which nanosilica is then extracted, and to study its properties. Also, the preparation of RHA has been optimized.

## Methods

### Materials

The raw material was obtained from a rice mill in the north of Iran. All reagents used were of analytical grade, and their solutions were made up in twice distilled, deionized water. HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, CH<sub>3</sub>COOH, NaOH, NH<sub>4</sub>OH, EDTA, KOH, and CaO were of laboratory grade from Merck (Merck & Co., Inc., Whitehouse Station, NJ, USA).

### Methods

#### Washing and acid treatment

RH was washed thoroughly with water to remove the soluble particles, dust, and other contaminants present, whereby the heavy impurities such as sand are also removed. It was then dried in an air oven at about 110°C for 24 h. The dried RH was refluxed with an acidic solution (HCl, HNO<sub>3</sub>, and CH<sub>3</sub>COOH in different concentrations) for nearly 90 min by stirring frequently. It was cooled and kept intact for about 20 h. It was then decanted and thoroughly washed with warm distilled water until the rinse became free from acid, and this was designated as RH'. The wet RH' was subsequently dried in an oven at 110°C for 24 h.

#### Thermal treatment

A weighed RH' as well as RH were subjected to heat treatment to obtain the ash. Samples were burned inside

a programmable furnace (Nabertherm controller B 170, Nabertherm GmbH, Lilienthal, Germany), and different methods were applied. Different temperatures (500°C, 700°C, and 1,000°C) and rates (2°C/min, 5°C/min, and 10°C/min) were checked. We designated these as ashes (RHAs).

#### Extraction of silica

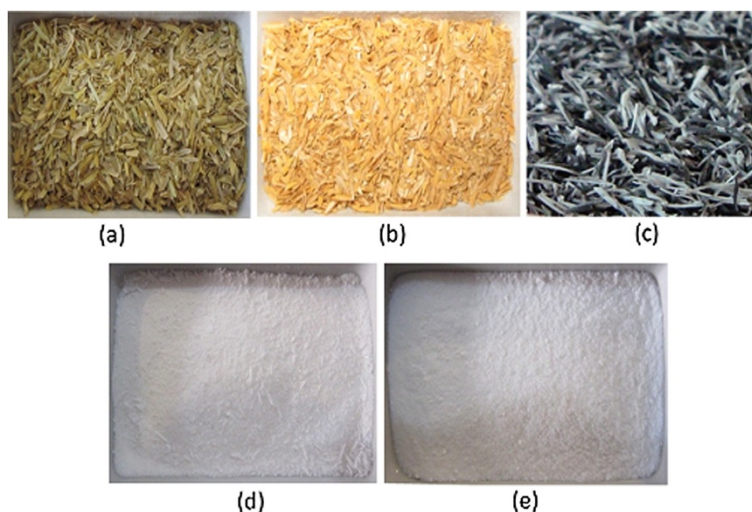
A sample of 20.0 g RHA was stirred in a 160-mL, 2.5-M sodium hydroxide solution. The solution was heated in a covered beaker for 3 h by stirring constantly and filtered; the residue was then washed with 40 mL of boiling distilled water. The obtained viscous, transparent, and colorless solution was allowed to cool down to room temperature, and 10 M H<sub>2</sub>SO<sub>4</sub> was then added under constant stirring at controlled conditions until it reached pH 2; NH<sub>4</sub>OH was added up to pH 8.5 and was allowed to stand at room temperature for 3 h.

#### Preparation of nanosilica

Nanosilica was prepared by reflux technique of the above extracted silica with 6.0 M HCl for 4 h and then washed repeatedly using deionized water to make it acid free. It was then dissolved in 2.5 M sodium hydroxide by stirring. H<sub>2</sub>SO<sub>4</sub> was added until it reached pH 8. The precipitate silica was washed repeatedly with warm, deionized water to make it alkali free and then dried at 50°C for 48 h in the oven.

#### Characterization

The silica content and the amount of metallic impurities in the samples were estimated by XRF Philips X-ray spectrometer (model PW 2404, Philips, Amsterdam, The Netherlands). Carbon, hydrogen, and nitrogen (CHN)



**Figure 1** Different images of RH in different conditions. (a) RH, (b) RH', (c) RHA with combustion temperature of 500°C, (d) RHA with combustion temperature of 700°C, and (e) nanosilica.

compositions were measured by HEKAtech elemental analysis (model Euro EA 3000, HEKAtech GmbH, Wegberg, Germany). Fourier transform infrared spectroscopy (FTIR) spectra were recorded with KBr pellets using a WQF-510 FTIR Rayleigh (Beijing Rayleigh Analytical Instruments Co., Ltd., Beijing, China). The X-ray diffraction (XRD) pattern of the sample was recorded on a Philips X'Pert (40 kV, 30 mA) X-ray diffractometer (Philips, Amsterdam, The Netherlands). Scans were taken with a  $2\theta$  and a counting time of 1.0 s using a Cu-K $\alpha$  radiation source ( $\lambda = 1.542 \text{ \AA}$ ) and a nickel filter. The surface areas were calculated from the linear part of the Brunauer-Emmett-Teller (BET) plot. Low-temperature nitrogen adsorption experiments were performed using a Quantachrome instrument (model Nova 2000, Quantachrome Instruments, Boynton Beach, FL, USA) system for measuring the surface area and pore volume. Transmission electron microscopy (TEM) examination was performed using a TEM microscope Philips CM 120 kV (Philips, Amsterdam, The Netherlands). The RH samples were heated in a programmable furnace (Nabertherm controller B 170, Nabertherm GmbH, Lilienthal, Germany). The potential variation was measured with a Jenway 3510 pH meter (Jenway, Staffordshire, UK) and a double-junction electrode.

#### Acidity measurement

For potentiometric titration, 0.05 g of solid was suspended in acetonitrile (90 mL) and stirred for 3 h. The suspension was titrated with a 0.05-M solution of *n*-butylamine in acetonitrile. The potential variation was measured using a double-junction electrode.

#### Lime reactivity

The reactivity of the ash was determined according to a given chemical method [23]. Lime reactivity of the various ashes was determined by suspending a small quantity

**Table 2 Lime reactivity values of RHA samples prepared at different conditions**

Entry	Type of acid	Concentration (M)	Temperature (°C)	Rate of heating (°C/min)	Lime reactivity (mg CaO/g)
1	HCl	0.01	700	5	55.23
2	HCl	0.1	700	5	58.72
3	HCl	1.0	700	5	62.71
4	HNO <sub>3</sub>	0.01	700	5	54.15
5	HNO <sub>3</sub>	0.1	700	5	57.41
6	HNO <sub>3</sub>	1.0	700	5	61.39
7	HOAc	0.1	700	5	50.81
8	HOAc	1.0	700	5	55.30
9	HOAc	5.0	700	5	59.75
10	HCl	0.1	500	5	47.82
11	HCl	1.0	500	5	51.56
12	HCl	0.1	1,000	5	56.21
13	HCl	1.0	1,000	5	58.16
14	HCl	0.1	700	2	50.06
15	HCl	1.0	700	2	59.42
16	HCl	0.1	700	10	60.72
17	HCl	1.0	700	10	64.27
18	H <sub>2</sub> O	-	700	5	56.16
19	No acid	-	700	5	37.73
20	RH	-	-	-	12.00

(approximately 0.3 g) of the ash in 50 mL of saturated lime solution at room temperature and by stirring after 18 h. The unused Ca was estimated by titration with a 0.001-M EDTA solution.

#### Results and discussion

The metallic ingredients have a substantial effect on the quality of silica from RH, which is mainly potassium that

**Table 1 Specific surface area and pore volume of RHA produced at different conditions**

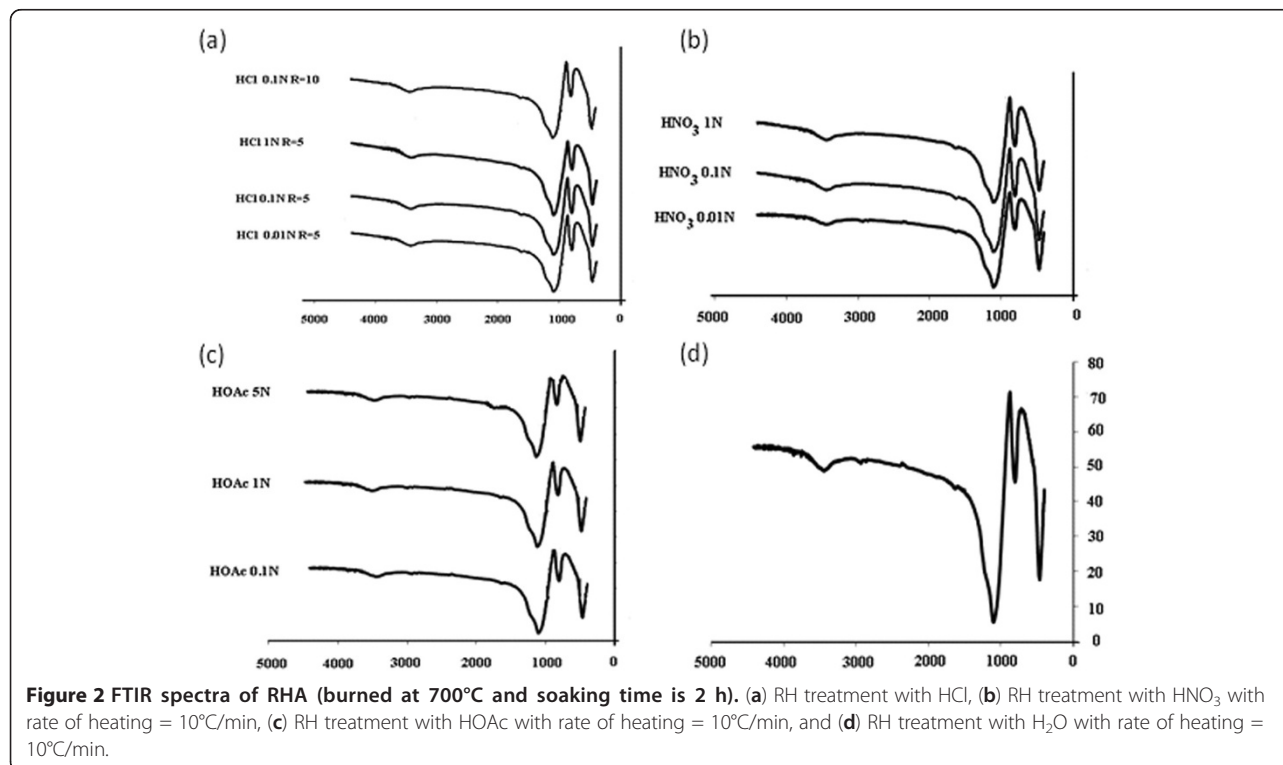
Entry	Type of acid	Concentration (M)	Rate of heating (°C/min)	Temperature (°C)	Surface area (m <sup>2</sup> /g)	Pore volume (cm <sup>3</sup> /g)
1	HCl	0.01	5	700	111.60	0.00548
2	HCl	0.1	5	700	139.80	0.00525
3	HCl	1.0	5	700	151.20	0.00610
4	HCl	1.0	10	700	153.10	0.00650
5	HNO <sub>3</sub>	0.1	5	700	129.80	0.00610
6	HOAc	0.1	5	700	13.40	0.00567
7	HCl	0.1	2	700	103.20	0.00498
8	HCl	0.1	10	700	148.70	0.00602
9	HCl	0.1	5	500	109.40	0.00587
10	HCl	0.1	5	1,000	98.41	0.00532
11	H <sub>2</sub> O	-	5	700	108.80	0.00600

**Table 3 Lime reactivity and chemical composition by X-ray fluorescence analysis**

	RHA	Extracted silica	Nanosilica
Color	White	White	White
SiO <sub>2</sub>	90.469	96.529	98.801
Na <sub>2</sub> O	0.630	Trace	Trace
K <sub>2</sub> O	Trace	Trace	Trace
MgO	0.050	0.038	0.020
CaO	Trace	Trace	Trace
Fe <sub>2</sub> O <sub>3</sub>	Trace	Trace	Trace
Al <sub>2</sub> O <sub>3</sub>	0.097	Trace	Trace
SO <sub>3</sub>	0.153	Trace	Trace
LOI	8.600	2.333	1.179
Lime reactivity (mg CaO/g)	64.27	65.50	72.50

causes surface melting and accelerates the crystallization of amorphous silica and carbon fixation in RHA. Also, a strong interaction occurs between the metallic ions and silica which leads to a considerable decrease in the surface area. Therefore, it is preferable to treat RH with an optimally acidic solution (such as HCl, HNO<sub>3</sub>, or CH<sub>3</sub>COOH in varying concentrations) to effectively diminish impurities and to obtain highly purified silica powder. The decreased impurities result mainly from chemical reaction between acid and metals, and then the reacted metals are leached from the acidic solution during filtration. The change in color during digestion is

also remarkable. The original brown color of the RH changes to yellowish and light yellow on increasing the time of digestion. Figure 1a,b shows the color of RH and RH' (after leaching with 0.1 M HCl for 90 min). The metals are also probably carried out from the volatiles during thermal decomposition. When burned, RH gives ash (RHA). If it is burned under controlled conditions, amorphous silica is produced, which is highly reactive in nature. The quality of RHA is dependent on various factors such as ashing temperature, time, rate of heating, and soaking time. The BET surface areas and pore volume of the produced RHA in different conditions are given in Table 1. The effect of different chemical treatments for the removal of metallic ingredients shows that leaching with 1.0 M HCl is the best one for high surface area and pore volume. It should be noted that the RHA formed at a lower combustion temperature (500°C) showed higher impurity and that at a higher temperature of combustion (1,000°C), the crystallization of silica increased. Also, the specific surface area decreased with increasing temperature (Table 1). This decrease of  $S_{BET}$  can be attributed to the portion of the pores destroyed in the process. Amorphous silica of high purity and relatively high surface area was prepared at 700°C. Rapid heating of RH does not allow the oxidation of carbon before the surface melting occurs, resulting in a higher amount of black particles. A good rate of heating was 10°C/min; in this condition, carbon formation and its oxidation occur before dissociation of metal oxides and



**Figure 2 FTIR spectra of RHA (burned at 700°C and soaking time is 2 h). (a) RH treatment with HCl, (b) RH treatment with HNO<sub>3</sub> with rate of heating = 10°C/min, (c) RH treatment with HOAc with rate of heating = 10°C/min, and (d) RH treatment with H<sub>2</sub>O with rate of heating = 10°C/min.**

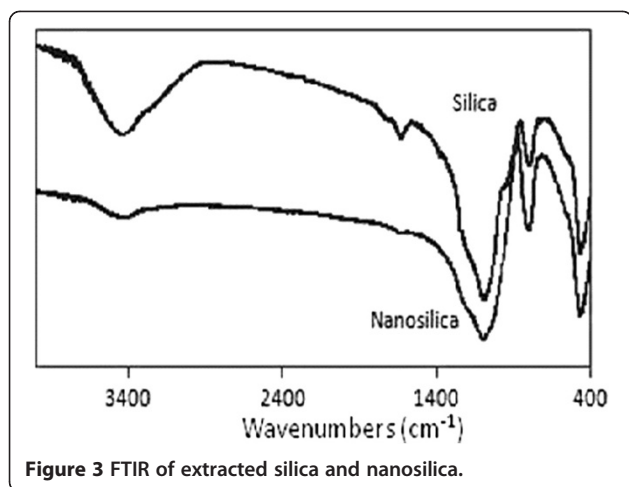
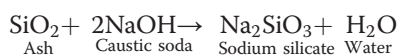


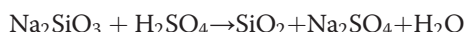
Figure 3 FTIR of extracted silica and nanosilica.

surface melting. Thus, the tendency of silica to retain carbon is eliminated, and white RHA is produced (Figure 1c,d). Lime reactivity was determined by a previously reported chemical method [23]. Table 2 gives the lime reactivity values of the ash samples prepared at different conditions. The RHA that was treated with 1.0 M HCl and burned at 700°C at the rate of 10°C/min and soaked in a programmable furnace for 2 h showed the maximum reactivity. Therefore, according to the results of Tables 1 and 2 and Figure 1, the above conditions are suitable for the preparation of highly purified RHA with high surface area. This white RHA was used for silica extraction and preparation of nanosilica.

Nanosilica was obtained from sodium silicate solution using precipitation method. The reaction is as follows:



The silica particles were generated from the solution by adding sulfuric acid as catalyst. The acidic condition of pH 2 indicates approximately the complete precipitation of silica from sodium silicate by the following reaction:



At a low silicate concentration and with the pH less than 8, Si(OH)<sub>4</sub> is the dominant species in its aqueous solution. At higher concentrations, the silanols, the Si(OH) groups, spontaneously polymerize to yield higher oligomers linked

Table 4 Content of carbon, hydrogen, and nitrogen of RH, RHA, and nanosilica at optimum conditions

	RH	RHA	Nanosilica
C (%)	25.313	1.285	0.119
H (%)	3.952	0.528	0.113
N (%)	0.551	0.000	0.000

Table 5 Properties of RHA and nanosilica

	RHA	Nanosilica
S <sub>BET</sub> (m <sup>2</sup> /g)	153.1	623
Pore volume (cm <sup>3</sup> /g)	0.00650	0.02408
Particle sizes (nm)	Approximately 60 <sup>a</sup>	6
Amorphous form (%)	97.75	99.90

<sup>a</sup>From XRD.

by a disiloxo bond. Such a reaction is most favorable when one of the silanols is deprotonated to a Si-O<sup>-</sup> group. These oligomers grow into colloid-sized silica particles in which larger particles grow at the expense of smaller ones. At a higher pH (greater than 8), however, the much more concentrated silicate solution is stable. This is because disiloxo bonds undergo nucleophilic attacks by OH<sup>-</sup> via a five coordinated intermediate.

### Characterization

After acid treatment and controlled combustion, 22.5% of RHA was produced. The percentage of pure silica in the RHA, extracted silica, and nanosilica were 90.469%, 96.529%, and 98.801%, respectively (Table 3). It is interesting to note that only MgO was detected in the extracted silica, and it was also present in a lower concentration in the nanosilica. Other impurities could be detected below the limit of quantification <0.01% (or 0.1 mg/g). The color of both RHA and nanosilica was white (Figure 1d,e).

Chemical composition (wt.%) as estimated after treatment of RH with 1.0 M HCl, combustion at 700°C at rate of 10°C/min, and after 2 h of soaking. Also, traces of P, Cl, Ni, Cu, Zn, Pd, W, and Bi were found in RHA and extracted silica

The FTIR spectra of almost all RHA samples gave typical bands of O-Si-O stretching (at 1,096 and 798 cm<sup>-1</sup>)

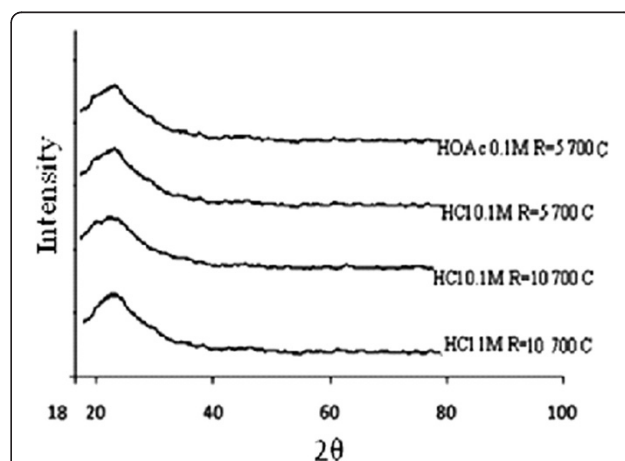
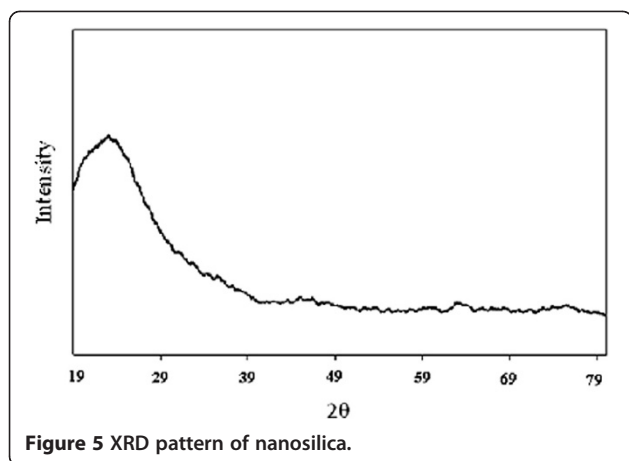


Figure 4 XRD pattern of some RHA samples with combustion temperature = 700°C and 2-h soaking time.



and bending vibrations (at  $466\text{ cm}^{-1}$ ). The bands at  $3,437$  and  $1,633\text{ cm}^{-1}$  corresponded to the O-H stretching and bending vibrations (Figure 2). Figure 3 shows the results of the FTIR analyses of extracted silica and nanosilica. The peaks at  $1,101$  and  $804\text{ cm}^{-1}$  are due to the Si-O-Si asymmetric and symmetric stretching modes, respectively. The band centered at  $469\text{ cm}^{-1}$  is due to the bending frequency of Si-O-Si. A large broad band around  $3,429\text{ cm}^{-1}$  is attributed to the presence of the O-H stretching frequency for the silanol group and the remaining adsorbed water. A band around  $1,630\text{ cm}^{-1}$  is assigned to the bending vibration of water molecules bound to the silica matrix. No peak was found between  $2,800$  and  $3,000\text{ cm}^{-1}$ . It means that there were no original organic compounds in the silica after controlled combustion and extraction. This result corresponds well with the carbon measurement by CHN analysis reported in Table 4. The FTIR spectra showed no significant changes in the peak position for extracted silica and nanosilica.

Silica with a high surface area is a good compound to be used as adsorbent or support for catalysts. Also, the reactivity of silica is directly related to its surface area. The BET-specific surface area and pore volume of RHA (at optimized conditions) and nanosilica are given in

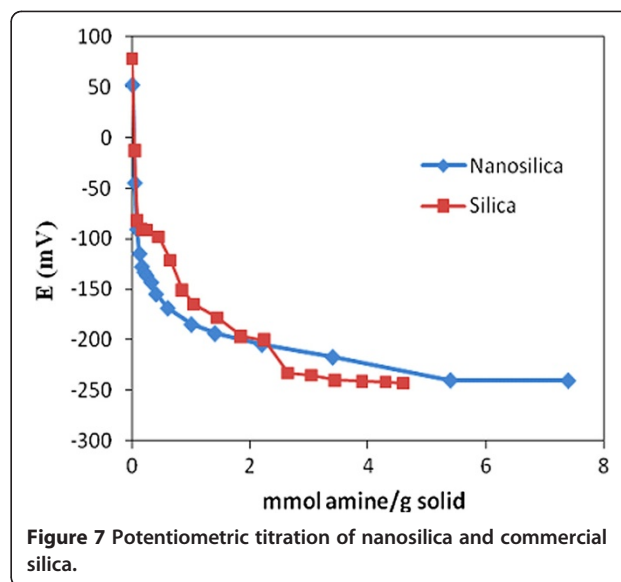
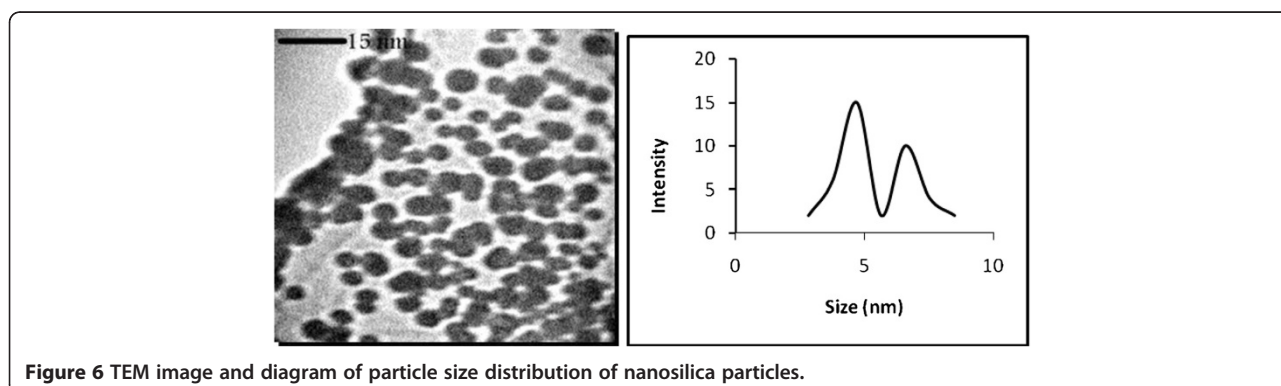


Table 5. A major contribution to the high surface area is that the organic matter has been broken up during the thermal decomposition of RHA, thus leaving a highly porous structure. The increasing pore volume in nanosilica confirms this phenomenon.

The evaluation of the amount of amorphous form of silica is very important. For this purpose, there are some specific methods in the literature [24,25]. One of them established that the degree of amorphous form of silica is estimated by the 'silica activity index,' which is determined by calculating the percentage of valuable silica dissolved in an excess of boiling 4 M KOH in a 3-min extraction period [24]. This method was investigated for extracted silica and nanosilica, and the results were 99.7% and 99.9% amorphous form, respectively. For clarification, the XRD results were examined. Figures 4 and 5 show, from some samples of RHA prepared at different conditions and nanosilica present, a broad peak at  $2\theta = 22^\circ$  associated with amorphous silica. The lack of sharp peaks indicates the absence of any ordered crystalline structure, confirming the results from the



silica activity index and the lack of crystalline phase. Also, there are no peaks of carbon, porous or activated, which can be produced by heating RH at a relatively lower temperature when this phenomenon occurs due to the presence of potassium [26]. However, an acid treatment of the husk removed most of the potassium, and the ash became free of fixed carbon. The size of the particles was estimated by the half width of the peak ( $\beta$ ) using Scherer's formula:

$$D = \frac{K\lambda}{\beta \cos\theta}$$

with  $K = 0.9$  nm as a constant,  $\lambda = 1.542$  Å as wavelength of Cu-K $\alpha$ , and  $\beta$  in radian. By a manual evaluation of the XRD chart, the particle size of nanosilica was found to be about 7 nm. Also, Figure 6 depicts the TEM micrograph of the nanosilica. The shape of the silica grains is spherical with an average particle size of 6 nm, which is in agreement with the size estimated by Scherer's formula.

The milligrams of CaO to grams of nanosilica and RHA are represented in Table 3 for the comparison of lime reactivity. Lime reactivity of nanosilica is relatively high, indicating that the silica is reactive.

Acidity measurements of catalyst by means of potentiometric titration with *n*-butylamine were used to estimate its relative acid strength according to the two values: (1)  $E_i$  as initial potential and (2) number of acid sites (Figure 7). The initial electrode potential ( $E_i$ ) indicates the maximum strength of the acid sites, and the value from which the plateau is reached (millimole amine per gram of solid) indicates the total number of acid sites that are present in the titrated solid [27]. The acidic strength of the solids can be classified according to the following range:  $E_i > 100$  mV (very strong sites),  $0 < E_i < 100$  mV (strong sites),  $-100 < E_i < 0$  mV (weak sites), and  $E_i < -100$  mV (very weak sites). According to the potentiometric titration curve, both the commercial silica (Aerosil 300 silica) and the nanosilica presented strong acidic sites. However, nanosilica has a higher surface area and a higher number of surface acidic sites compared with commercial silica ( $n = 5.4$  and  $2.6$  mmol amine/g solid, respectively).

## Conclusions

Acid treatment of RH followed by thermal combustion under controlled conditions gave 22.50% ash of which 90.469% was silica. At optimized conditions, a nano-sized, highly purified silica (98.8 mass percentage) was produced with a high surface area, high reactivity, and 99.9% in amorphous form. This nanosilica showed strong and a large number of acidic sites in comparison with commercial silica, making it a good support for catalysts. Considering the availability of RH worldwide, this

study shows that economic technology can be used by rice-producing companies to produce highly effective silica and to reduce the environmental impact of the usually uncontrolled burning of the by-products of the industry, namely RH. This is particularly relevant to an agro-based country, which is still importing micro- and nanosilica for some applications.

## Competing interests

The authors declare that they have no competing interest.

## Authors' contributions

ER conceived the study and did the major part of the article with SS. MF carried out the X-ray diffractions. MS did some of the characterizations. All authors read and approved the final manuscript.

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